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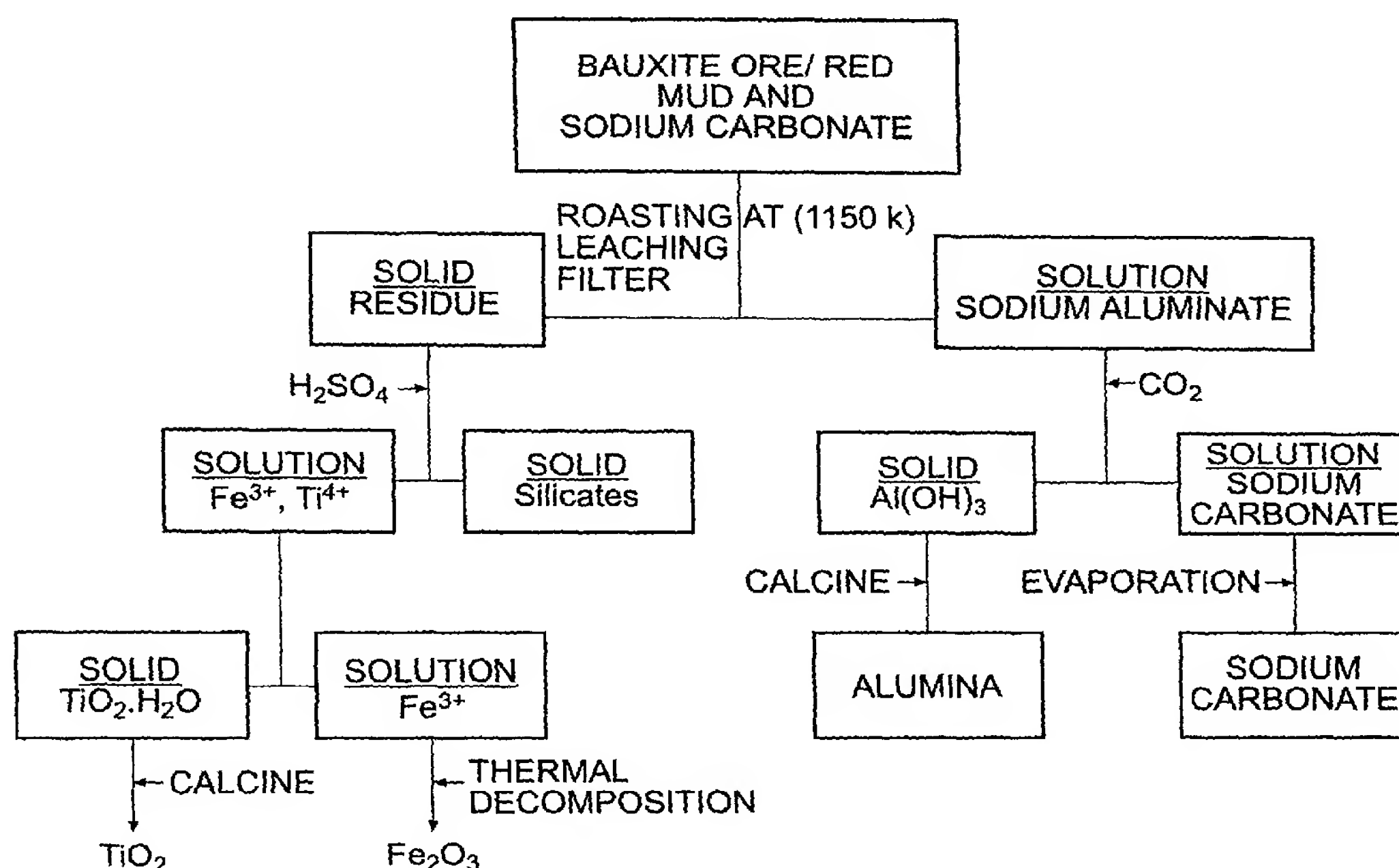
(43) International Publication Date  
7 February 2002 (07.02.2002)

PCT

(10) International Publication Number  
**WO 02/10068 A1**

- (51) International Patent Classification<sup>7</sup>: **C01F 7/08**, C01G 23/047, 49/06
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- (21) International Application Number: PCT/GB01/03370
- (22) International Filing Date: 30 July 2001 (30.07.2001)
- (25) Filing Language: English
- (26) Publication Language: English
- (30) Priority Data: 0018592.6 29 July 2000 (29.07.2000) GB
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- (81) Designated States (national): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.
- (84) Designated States (regional): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).
- Published:  
— with international search report
- For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: PRODUCTION OF METAL OXIDES



(57) Abstract: There is described a process for the purification of metal oxides which comprises the steps of: (i) roasting a mineral ore in the presence of an alkali; (ii) extracting the desired metal salt produced in step (i); (iii) precipitation of a hydroxide salt; and (iv) converting the hydroxide salt into an appropriate oxide. The process applies to the recovery of alumina, titania and ferric oxide from bauxite and/or red mud.

## Production of Metal Oxides

The present invention relates to a process for the production of metal oxides.

5 More particularly, the process relates to the production of iron, aluminium and titanium from aluminous minerals such as bauxite, alumina-rich clays and the solid waste generated from the Bayer's process called, the red mud. The process further involves roasting of the above mentioned materials with alkali, eg sodium or potassium carbonate or sodium or potassium hydroxide, mixed with the carbonates, in  
10 air and subsequent leaching by water and sulphuric acid.

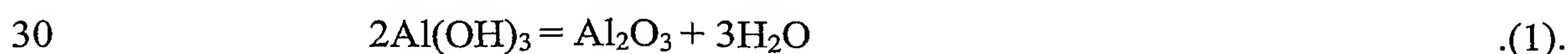
Aluminium is the most abundant metallic element in the earth's crust. It is normally found combined with other elements, and does not occur in the pure state. It appears in a wide variety of minerals combined with oxygen, silicon, the alkali and alkaline-  
15 earth metals and as hydroxides, sulphates and phosphates. The bauxite is a sedimentary rock that contains economically recoverable quantities of the aluminium oxide minerals Gibbsite, Böhmite, and Diaspore.

The average chemical composition of major constituents of bauxite (2) in weight  
20 percentage is given below:

Al <sub>2</sub> O <sub>3</sub> *	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	Moisture (loss on ignition)
50 - 52 %	4-6%	16-18 %	2-3%	21 to 28%

25

Most of the Al<sub>2</sub>O<sub>3</sub> content is, in reality, present in the form of Gibbsite,  $\gamma$ -Al(OH)<sub>3</sub> or Böhmite,  $\alpha$ -AlO(OH). The hydroxide ions, upon ignition, are converted to water resulting in alumina via the reaction 1:



Similarly, in bauxite, iron is present as Goethite,  $\alpha$ -FeO(OH) and hematite,  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>;

titanium is present in bauxite as Anatase  $\text{TiO}_2$ ; and Silicon dioxide may occur either as quartz, or as complex silicates.

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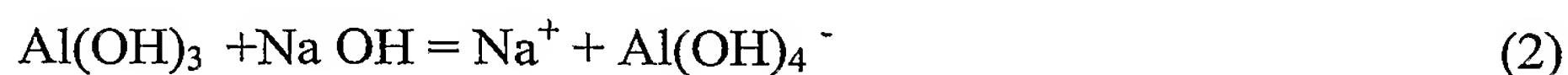
Bayer's process is widely used for the production of pure  $\text{Al}_2\text{O}_3$  from bauxite. In this process, bauxite is dissolved in an NaOH solution at 400 - 500K to produce Sodium aluminate which is  $(\text{NaAlO}_2)$ . The  $(\text{NaAlO}_2)$  formed is soluble in water and filtered out. Aluminium hydroxide  $(\text{Al}(\text{OH})_3)$  is then precipitated from the solution and the.

10  $\text{Al}(\text{OH})_3$  is calcined at 1350 to 1400K to produce pure alumina  $(\text{Al}_2\text{O}_3)^{[1]}$ . The unreacted constituents of bauxite are the oxides of iron, titanium and a fraction of unreacted alumina. These oxides form the residue from the process and it is called the red mud. A complete recovery of alumina is economically not possible via the Bayer process.

15

The Bayer process begins with the preparations of bauxite by grinding to produce a uniform composition. In most alumina plant, the bauxite is ground while suspended in a portion of the Bayer process alkali solution. This slurry is then mixed with the remaining NaOH solution at 400K. The mixture, with hot NaOH solution is then

20 treated in a digester vessel at well above atmospheric pressure. The main reaction in the digester is given by equation 2:



25 The filtered solution containing sodium aluminate is cooled from 400 K down to 335 - 345K temperature range. The concentration at this temperature is such that the solution is saturated with  $\text{Al}(\text{OH})_3$ , but is not supersaturated to cause spontaneous crystallisation. A seed of aluminium hydroxide is added at this point to precipitate  $\text{Al}(\text{OH})_3$  from the sodium aluminate mother liquor. The aluminium hydroxide

30 precipitate is calcined in the temperature range of 1350 to 1400K for the production of pure  $\text{Al}_2\text{O}_3$  via the decomposition of aluminium hydroxide, shown in reaction 1.

However, one disadvantage of the Bayer process is that  $\text{Al}_2\text{O}_3$  is not completely removed from the bauxite ore. A fraction of alumina always remains in the solid filter residue, this is known as "the red mud". Furthermore, the other major constituents of bauxite, e.g. iron and titanium, remain as complex oxides in the red mud.

5

The red mud is highly alkaline ( $\text{pH}=10-12$ ) and creates a substantial environmental hazard. Its safe disposal and utilisation thus poses a major scientific challenge (3). The red mud also has a high  $\text{Al}_2\text{O}_3$  content. The average chemical composition of the constituents of red mud is:-

10

$\text{Al}_2\text{O}_3$  (23 –30 wt%),  $\text{SiO}_2$  (8 wt%),  $\text{Fe}_2\text{O}_3$  (35–50 wt%), and  $\text{TiO}_2$  (15-18 wt%).

15

A micrograph of red mud sample is shown in figure 1. It mainly consists of three phases as marked A, B, and C in the micrograph. Phase A is rich in aluminium, whereas phase B and phase C are rich in iron and titanium respectively. Electron Diffraction X-Ray (EDX) analysis of the three phases can be seen in figures 2 to 4. An X-ray diffraction pattern of dry red mud is shown in figure 5. Therefore, from the X-ray diffraction and EDX analysis, it is apparent that alumina, silica, iron oxide, and titanium dioxide are present in complex mineralogical forms in red mud.

20

The most important environmental problem in the Bayer process is the safe disposal of red mud. The solution left with the residue after washing is still very alkaline and it cannot be allowed to contaminate the ground water. An aqueous slurry of the residue that had been washed with 1000 times its mass of distilled water still reaches a pH value of 10.5. The fine residue, even after years of consolidation, does not have enough strength to support buildings and equipment. These properties make disposal of red mud a difficult problem (3). In many places red mud is disposed at sea where the alkalinity was diluted by large quantities of water. This method of red mud disposal was employed in Europe and Japan in the past, whereas in the United States the red mud was disposed via the inland waterways. Today, however, due to increased environmental awareness and need to reduce the threat to marine and aquatic life, any

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new alumina plant will have to meet environmental specifications for disposing of red mud.

Therefore, in view of the increasing problem of waste disposal, it is desirable to  
5 recover the oxides of iron, titanium and the remaining fraction of aluminium from the red mud, thereby obviating the need for waste disposal.

Accordingly, the object of the present invention is to provide a process, which enables the recovery of, particularly, unreacted  $\text{Al}_2\text{O}_3$ , and  $\text{TiO}_2$  from bauxite and/or from the  
10 process residue, red mud. The process offered by the present invention advantageous because, *inter alia*, it provides an opportunity for the recovery of minerals from red mud, but also it has been found that the above oxides and hydroxides could be extracted more efficiently, not only from bauxite, but also from alumina-rich clays and/or red mud via the process of the invention.

15 Thus, the present invention provides an alternative and more environmentally acceptable process for the extraction of aluminium oxide and oxides of iron and titanium from, red mud, bauxite and/or alumina-rich clays.

20 Thus, according to the invention we provide a process for the purification of metal oxides which comprises the steps of;

- (i) roasting a mineral ore in the presence of an alkali;
- (ii) extracting the desired metal salt produced in step (i);
- 25 (iii) precipitation of a hydroxide salt; and
- (iv) converting the hydroxide salt into an appropriate oxide.

More particularly, the process comprises the steps of;

- 30 (i) roasting a mineral ore (bauxite/clay) and alumina-containing residues in the presence of an alkali and/or alkali mixture;



- (ii) extracting the desired metal salt produced in step (i) using an aqueous and/or a dilute ammoniacal solution in aqueous media to separate water-soluble alkali aluminate from undigested metal oxide filter residue;
- 5 (iii) precipitation of aluminium hydroxide from alkali aluminate solution by bubbling CO<sub>2</sub> gas or oxalic acid medium for maintaining a constant pH;
- (iv) calcining aluminium hydroxide formed in step (iii).
- 10 The actual temperature used in step (i) of the process of the invention may vary, depending, *inter alia*, upon the nature of the mineral ore, the nature of the metal oxide to be isolated, etc. Preferentially, the temperature used in step (i) of the process of the invention is greater than 400K, preferably greater than 500K, more preferably between 600 and 1300K, especially between 700 to 1200K, e.g. at 1150K. We have
- 15 surprisingly found that 1150K is the optimum temperature for the isolation of aluminium, although variations on this temperature, within the ranges identified herein, will also produce satisfactory results.

The process of the invention is suitable for use in purification of mineral ores, e.g.

20 bauxite. However, as already described, the process is also suitable for the purification of waste materials, such as red mud. Thus, it should be understood that the phrase "mineral ore" when used in the definition of the invention herein should be construed as including alumina-rich clays, waste materials from other processes and in particular red mud produced from the Bayer process.

25

The alkali used in step(i) of the process of the invention is preferentially a carbonate which may comprise any conventionally known carbonate or a mixture of carbonates. Especially preferred carbonates are the carbonates of the Group Ia and Group IIa metals, i.e. the alkali or alkaline earth metals. Such metal salts therefore include, for

30 example, lithium, sodium, potassium, magnesium and/or calcium carbonates. Carbonates of the alkali metals are most preferred, for example sodium or potassium

carbonate. Most preferentially sodium carbonate is used. Alternatively, a mixture of a hydroxide, eg sodium or potassium hydroxide, and a carbonate may be used.

5 The extraction process of step (ii) may use a variety of solvents or mixtures of solvents. The solvent may be varied depending upon the nature of metal salt being isolated. However, preferentially, the solvent will be one which can readily be disposed of with minimal damage to the environment. Thus, the most preferred solvent is water, usually hot water above 25°C. A dilute solution of ammonium salts in water is recommended for enhancing the extraction of alkali aluminate. The  
10 treatment of calcined minerals with water or with dilute ammonical solution enhances the efficiency of alkali aluminate extraction from the roasted minerals.

The precipitation of aluminium hydroxide,  $\text{Al}(\text{OH})_3$  from alkali aluminate solution is initiated by passing  $\text{CO}_2$  or oxalic acid solution. The precipitated aluminium  
15 hydroxide is dried and calcined at 1400K for producing alumina powder as it is done in the Bayer process.

In the acidification process of step (iii) any conventionally known acids may be used, including organic and inorganic acids. Alternatively mixtures of acids may be used.  
20 Inorganic acids are preferred and such acids may be selected from, but are not limited to, hydrofluoric acid, hydrochloric acid, nitric acid, sulphuric acid and an acidic oxide or mixtures thereof. When the metal ore being isolated is aluminium, then an acidic oxide is preferred. The most preferred acidic oxide is carbon dioxide.

25 In step (iv) of the process of the invention, the conversion of the hydroxide salt to the oxide end product may comprise any conventionally known oxidation step, such steps may include thermal decomposition, calcining, etc. When the metal oxide is alumina, then the oxidation step preferentially comprises calcining.

30 The material flow sheet is similar to the Bayer process, except the  $\text{NaAlO}_2$  formation is carried out at an elevated temperature above 1050 K in the absence of water and in the presence of sodium carbonate. In the present process, bauxite ore or red mud is

homogeneously mixed with the stoichiometric amount of sodium carbonate required to convert alumina to sodium aluminate. The mixture is roasted above 1050K in air. The reactions which take place above 1050K are:



The roasted mass contains water soluble sodium aluminate and other insoluble components of ore. The roast product is extracted with hot water so that all of the sodium aluminate phase dissolves in water. The filtrate consists of the water-soluble sodium aluminate, whereas the hydroxides of iron, titanium oxide and other impurities remain present in the solid residue, which is part of the unreacted bauxite. A micrograph of the solid residue calcined at 1400K is shown in figure 6. EDX analysis of the two phases as marked B and C is given in figures 7 and 8. The phase B is rich in iron, whereas the phase C is comparatively richer in titanium. The presence of aluminium oxide is not detected in the EDX analysis, as its concentration appears to be well below 5 wt%. Alumina is completely extracted from the red mud as sodium aluminate, which is converted to  $\text{Al}(\text{OH})_3$  via the acidification with carbon dioxide gas.

20



$\text{Al}(\text{OH})_3$  precipitate is then filtered and calcined at 1350-1400K to produce pure  $\text{Al}_2\text{O}_3$ . The micrographs and X-ray powder diffraction of the alumina extracted from the red mud and bauxite are shown in figures 9a and 9b respectively. The phases identified in the powder diffraction pattern show the evidence for the dominant  $\alpha$  and  $\epsilon$  types of alumina. The grains are well defined and clean. In Figures 10a and 10b, the particle size distribution of dried and calcined aluminium hydroxide and alumina are shown respectively. The alumina particle size distribution shown in Figure 10b is bimodal, which can be altered by varying the time and temperature of calcination. The filtrate containing  $\text{Na}_2\text{CO}_3$  is evaporated to regenerate sodium carbonate. Production of soda is one of the main advantages of this new process.

30



The residue containing iron and titanium is mixed with 98%  $\text{H}_2\text{SO}_4$  so as to make a slurry. The ratio of  $\text{H}_2\text{SO}_4$  to residue is chosen such that the weight ratio of  $\text{H}_2\text{SO}_4$  to  $\text{TiO}_2$  in the suspension produced by the hydrolysis is between 2-2.5. The slurry  
5 obtained by digestion is dissolved in cold water or in dilute aqueous sulphuric acid ( $\text{H}_2\text{SO}_4$ ). Undissolved solid material containing silicates is removed completely by filtration. Titanium oxide hydrate is precipitated from the filtered solution by hydrolysis at 375 -390K. The hydrate is filtered off from the solution and washed with water or dilute acids to remove dissolved sulphates of other metallic impurities  
10 adhered on to the precipitate surface. Finally the hydrate is calcined in the temperature range of 1050 to 1300K to produce pure titanium oxide, which can be used for manufacturing pigments.

The solution containing iron salts are concentrated and then thermally decomposed to  
15 form iron oxide and sulphur dioxide. Alternatively, the filtrate can also be evaporated to obtain  $\text{FeSO}_4$ , which can be used for water purification.

According to a further aspect of the invention we provide a metal oxide produced by a process as hereinbefore described. Thus, the metal oxide produced may be selected  
20 from an aluminium oxide, an iron oxide and a titanium oxide. Preferably, the metal oxide produced is selected from  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$  and  $\text{TiO}_2$ ; most preferably the metal oxide produced is  $\text{Al}_2\text{O}_3$ .

The process of the present invention is advantageous, *inter alia*, because alumina  
25 present in bauxite and red mud was extracted with 98 % efficiency via the alkali-roasting process by forming sodium or potassium aluminate above 400K. Furthermore,  $\text{TiO}_2$  was recovered from the residue obtained after extracting aluminium by dissolving the residue in concentrated sulphuric acid and by subsequent hydrolysis and calcination. In addition,  $\text{Fe}_2\text{O}_3$  was recovered by  
30 precipitating  $\text{Fe}(\text{OH})_3$  from the filtrate by the addition of an alkali or thermal decomposition of the filtrate.

The filtrate residue derived from potassium carbonate/potash roasting of alumina containing minerals yield residues rich in  $K^+$  ions. Potassium ion containing residue can advantageously be used as a soil conditioner and for making fertiliser by mixing with phosphate residue, phospho-gypsum, and/or Basic Oxygen Furnace slag from steel making plant.

The new process of the invention has been developed for extracting alumina, iron oxide ( $Fe_2O_3$ ) and/or titanium oxide ( $TiO_2$ ) from bauxite and red mud. The process involves roasting of bauxite and/ or red mud with sodium carbonate ( $Na_2CO_3$ ) in air. The roasted mass is digested in water and filtered. The filtrate contains sodium aluminate.  $Al(OH)_3$  is precipitated by passing carbon dioxide ( $CO_2$ ) through the filtrate. Pure alumina is produced by calcining the  $Al(OH)_3$  precipitate. Residue is digested in concentrated sulphuric acid ( $H_2SO_4$ ) and diluted with water. Titanium oxide hydrate is precipitated via the hydrolysis of the acid solution at 375-390K. Iron oxide is recovered from the filtrate by evaporation and thermal decomposition. The  $CO_2$  generated from the decomposition of sodium carbonate can be recycled during the precipitation of  $Al(OH)_3$  from the  $NaAlO_2$  solution to reform  $Na_2CO_3$ . The recovery of other oxides,  $TiO_2$  and  $Fe_2O_3$  will yield a zero waste process for the manufacture of alumina.

20

The invention is illustrated by the following examples.

In which Figure 1 is a micrograph of red mud;

Figure 2 is an EDX analysis of an alumina-rich phase;

25

Figure 3 is an EDX analysis of an iron rich phase;

Figure 4 is an EDX analysis of a titanium rich phase;

Figure 5 is an X-ray diffraction pattern of dry red mud;

Figure 6 is a micrograph of red mud after alumina extraction;

Figure 7 is an EDX analysis of the iron rich phase after extracting alumina from red mud;

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Figure 8 is an EDX analysis of the titanium rich phase after extracting alumina from red mud;

Figure 9a is a micrograph of alumina extracted from red mud;

Figure 9b is an X-ray diffraction pattern of alumina extracted from red mud;

Figure 10a is a particle size distribution of  $\text{Al}(\text{OH})_3$  after drying;

Figure 10b is a particle size distribution of alumina after calcining at 1400K;

5 and

Figure 11 is a schematic representation of a process of the invention.

### EXAMPLE I: Bauxite ore from Ghana

10 The extraction efficiency is defined by  $\psi$  in percentage, which is dependent on the concentration of aluminium in the ore and in the residue.

$$\psi = \frac{[\%Al]_{ORE} - [\%Al]_{residue}}{[\%Al]_{Ore}} \times 100 \quad ..(5)$$

15 The following inventive steps involved in achieving nearly 100% extraction of alumina.

1. Bauxite ore from Ghana (approximate composition: 55% of  $\text{Al}_2\text{O}_3$ , 12 % of  $\text{Fe}_2\text{O}_3$ , 2% of  $\text{TiO}_2$ , 2 % of  $\text{SiO}_2$ , and moisture) was homogeneously mixed with  
20 sodium carbonate in the ratio 1: 0.60 .
2. The mixture was heated at 1150°C for 2 hours in an electrically heated furnace in air.
- 25 3. The reaction product was digested in hot water and filtered.
4. The filtrate was acidified with carbon dioxide to convert water-soluble sodium aluminate to  $\text{Al}(\text{OH})_3$  precipitate.
- 30 5.  $\text{Al}(\text{OH})_3$  was filtered out and calcined to produce pure  $\text{Al}_2\text{O}_3$ . The extraction efficiency of alumina was nearly 98%.

6. The filtrate was evaporated and sodium carbonate was recovered.
7. The residue containing iron oxide ( $\text{Fe}_2\text{O}_3$ ) and titanium dioxide ( $\text{TiO}_2$ ) was mixed  
5 with 98%  $\text{H}_2\text{SO}_4$  for making a slurry. The ratio of  $\text{H}_2\text{SO}_4$  to residue was chosen in  
such a way that the weight ratio of  $\text{H}_2\text{SO}_4$  to  $\text{TiO}_2$  in the suspension produced by  
the hydrolysis was maintained between 2 to 2.5.
8. The slurry obtained by digestion was dissolved in water.
- 10 9. Undissolved solid material containing silicates was removed completely by  
filtration.
10. Titanium oxide hydrate was precipitated from the filtered solution by hydrolysis in  
15 the temperature range 375 to 390K.
11. The titanium oxide hydrate was filtered off from the solution and calcined in the  
temperature range of 1050 to 1300K to produce pure titanium oxide.
- 20 12. The filtrate was treated with NaOH to precipitate Fe as  $\text{Fe}(\text{OH})_3$ . The precipitate  
was filtered out and calcined to produce  $\text{Fe}_2\text{O}_3$ .

## EXAMPLE II ( Red Mud)

- 25 Red mud from ALCAN (approximate composition: 46 %  $\text{Fe}_2\text{O}_3$ , 22 % of  $\text{Al}_2\text{O}_3$ , 8%  
of  $\text{TiO}_2$ , 8 % of  $\text{SiO}_2$ , 3-4 MgO and CaO and loss on ignition was 10-12 wt%) was  
homogeneously mixed with sodium carbonate in the ratio 1: 0.25. The experiments  
were carried out as described in the above examples by steps 2 to 12. The extraction  
efficiency for alumina was over 98 %.

30

The extraction efficiency of alumina from bauxite and red mud does not change when  
soda is replaced by potassium carbonate or potash. The red mud obtained in this case

contains alkali in the form of potassium ions and does not have harmful effect of soda in red mud, which cannot be used as a fertiliser or soil conditioner. Below all the claims are referred in terms of alkali roasting of red mud and bauxite for the extraction of alumina, titania, and iron oxides as by-product.

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*References:*

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## Claims

1. A process for the purification of metal oxides which comprises the steps of;
  - 5 (i) roasting a mineral ore in the presence of an alkali;
  - (ii) extracting the desired metal salt produced in step (i);
  - (iii) precipitation of a hydroxide salt; and
  - (iv) converting the hydroxide salt into an appropriate oxide.
- 10 2. A process according to Claim 1 characterised in that the roasting process of step (i) is carried out at an elevated temperature of greater than 400K.
3. A process according to Claim 2 characterised in that the roasting process of step (i) is carried out at a temperature of greater than 500K.
- 15 4. A process according to Claim 3 characterised in that the roasting process of step (i) is carried out at a temperature of between 600 and 1300K.
5. A process according to Claim 4 characterised in that the roasting process of step (i) is carried out at a temperature of between 700 to 1200K.
- 20 6. A process according to Claim 5 characterised in that the roasting process of step (i) is carried out at a temperature of 1150K.
- 25 7. A process according to Claim 1 characterised in that the mineral ore is bauxite.
8. A process according to Claim 1 characterised in that the mineral ore is red mud.
- 30 9. A process according to Claim 7 characterised in that the mineral ore is a mixture of bauxite and red mud.

10. A process according to Claim 1 characterised in that the carbonate is a carbonate of a Group Ia and Group IIa metals or a mixture thereof.
11. A process according to Claim 1 characterised in that the carbonate is an alkali  
5 metal carbonate or a mixture of alkali metal carbonates.
12. A process according to Claim 11 characterised in that the carbonate is selected from sodium carbonate and potassium carbonate.
- 10 13. A process according to Claim 12 characterised in that the carbonate is sodium carbonate.
14. A process according to Claim 12 characterised in that the carbonate is a mixture of sodium carbonate and potassium carbonate.
- 15 15. A process according to Claim 1 characterised in that the solvent used in the extraction process of step (ii) is water.
15. A process according to Claim 1 characterised in that the acid used in the  
20 acidification process of step (iii) is an inorganic acid.
16. A process according to Claim 15 characterised in that the inorganic acid is selected from hydrofluoric acid, hydrochloric acid, nitric acid, sulphuric acid and an acidic oxide or a mixture thereof.
- 25 17. A process according to Claim 16 characterised in that the acid is an acidic oxide.
18. A process according to Claim 17 characterised in that the acidic oxide is  
30 carbon dioxide.

19. A process according to Claim 1 characterised in that the process of step (iv) comprises calcining.
20. A process according to Claim 1 characterised in that the oxide produced is  
5  $\text{Al}_2\text{O}_3$ .
21. A process according to Claim 1 characterised in that the oxide produced is  $\text{Fe}_2\text{O}_3$ .
- 10 22. A process according to Claim 1 characterised in that the oxide produced is  $\text{TiO}_2$ .
23. A metal oxide produced by a process according to Claim 1.
- 15 24. A metal oxide according to Claim 23 characterised in that the metal is selected from aluminium, iron and titanium.
25. A metal oxide according to Claim 24 characterised in that the metal oxide is selected from  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$  and  $\text{TiO}_2$ .
- 20 26. A metal oxide according to Claim 25 characterised in that the metal oxide is  $\text{Al}_2\text{O}_3$ .
27. A metal oxide according to Claim 26 characterised in that the metal oxide is  
25  $\text{Fe}_2\text{O}_3$ .
28. A metal oxide according to Claim 27 characterised in that the metal oxide is  $\text{TiO}_2$ .
- 30 29. A process or a metal oxide substantially as described with reference to the accompanying examples.

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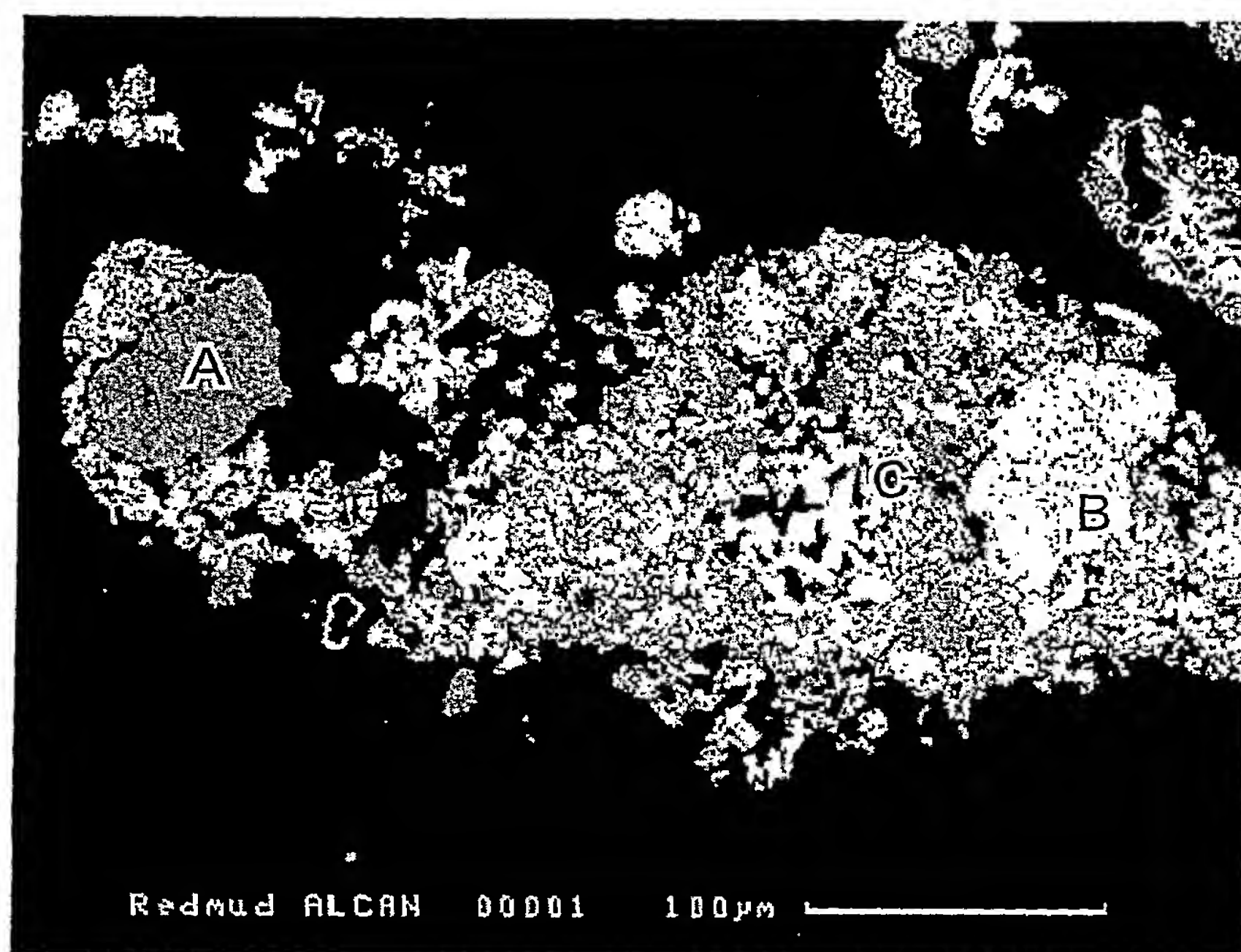


Fig. 1

Micrograph of ALCAN red mud

A : Alumina rich phase B : Iron rich phase C : Titanium rich phase

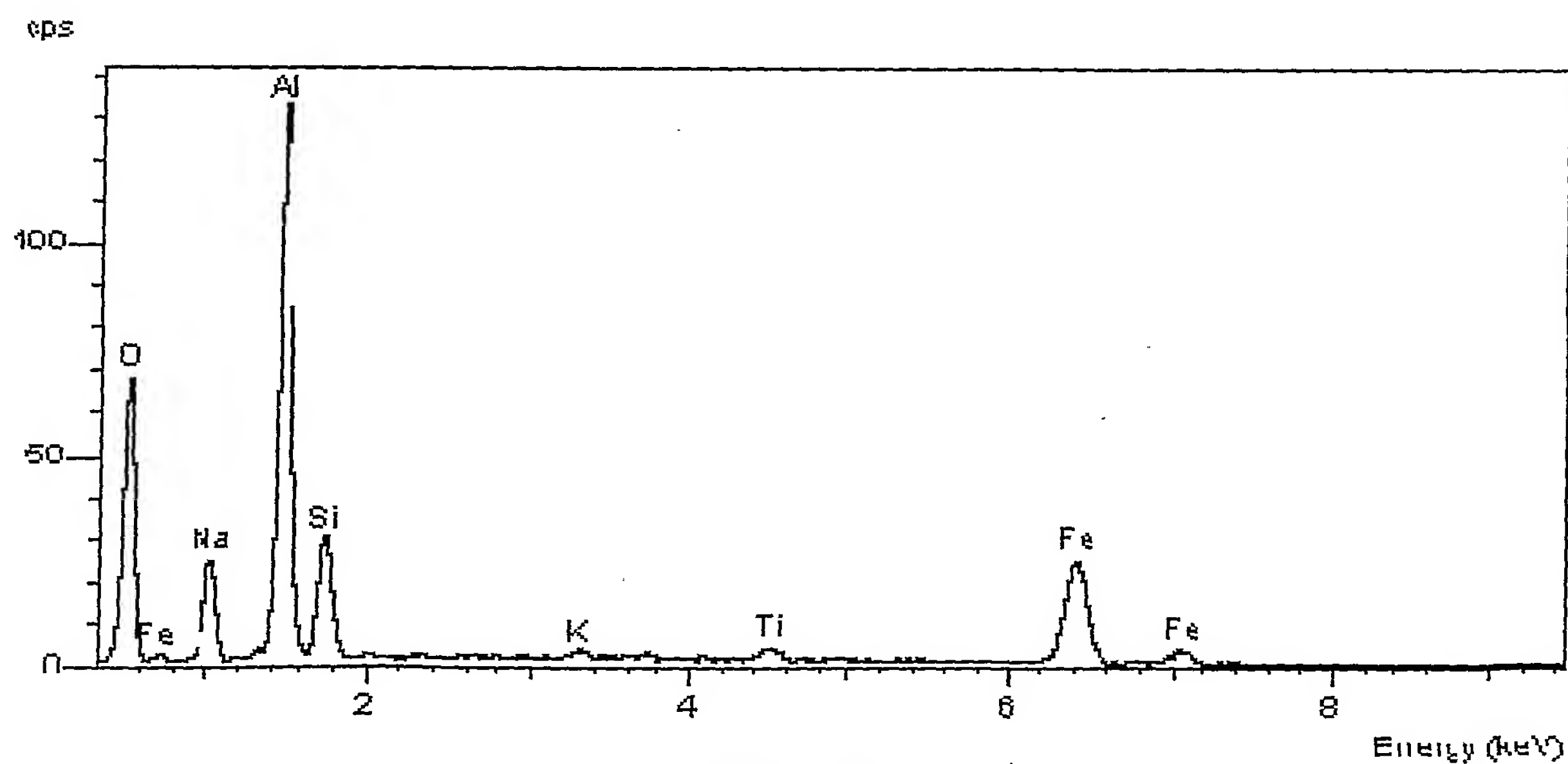


Fig. 2

EDX analysis of alumina rich phase (A) in red mud

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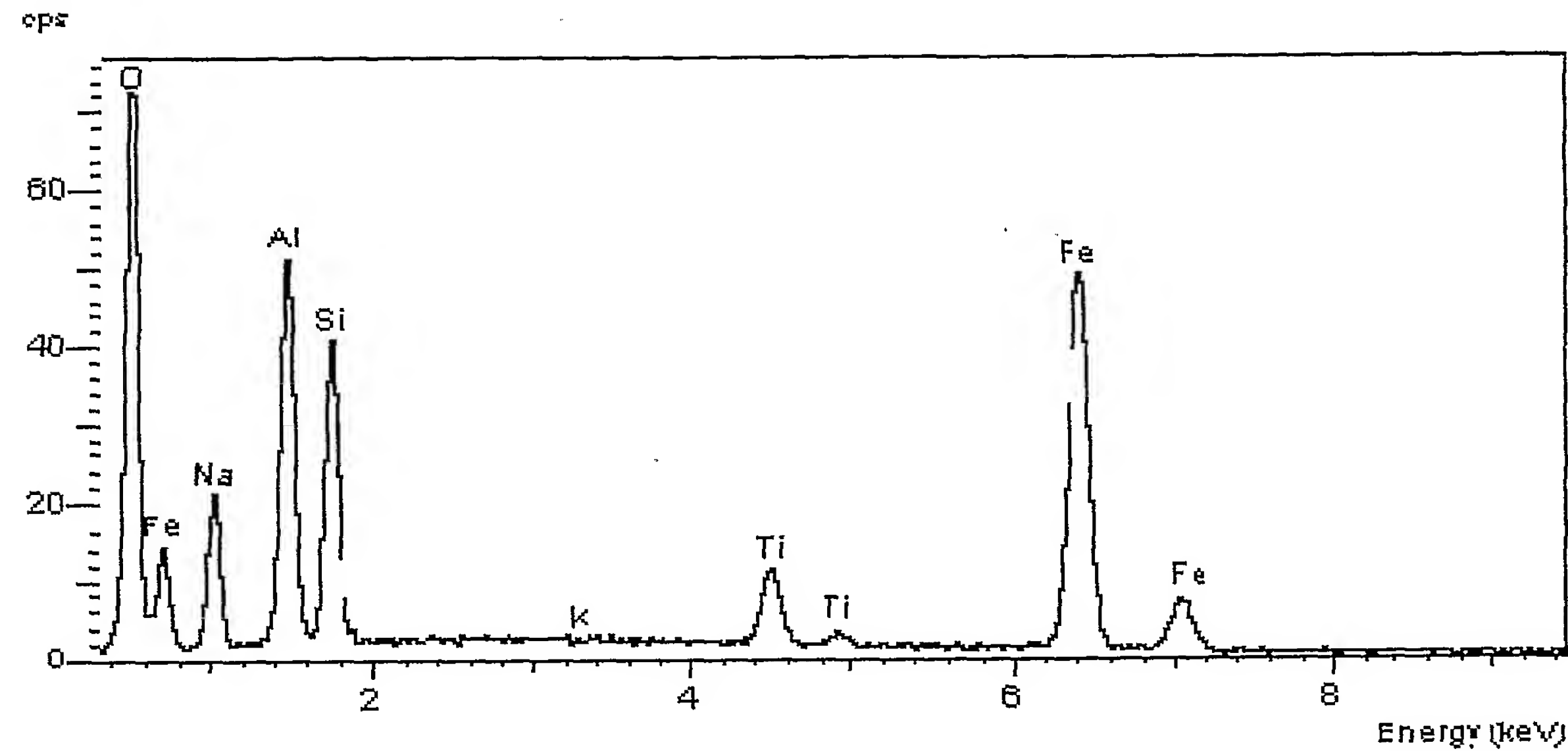


Fig. 3  
EDX analysis of iron-rich phase (B) in red mud

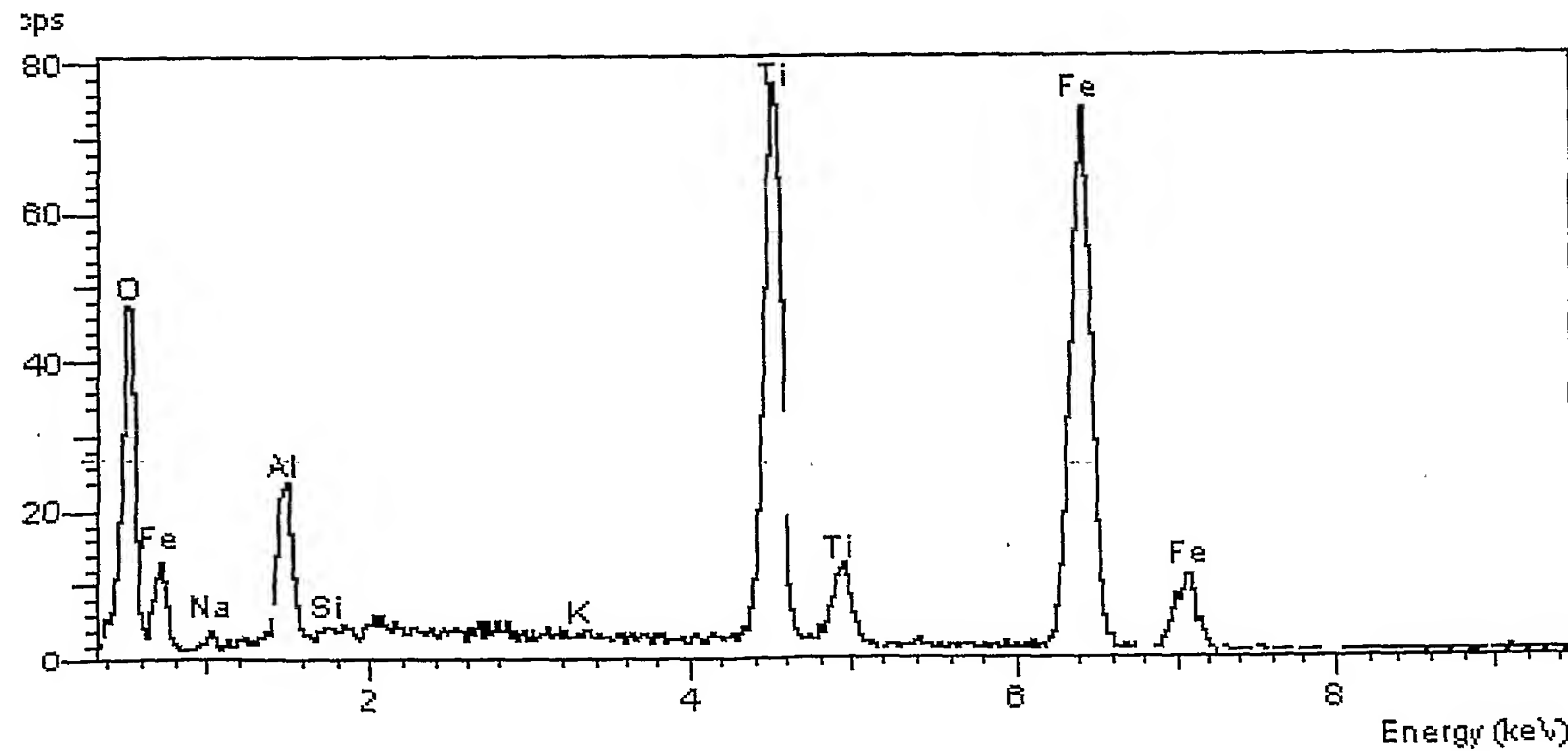


Fig. 4  
EDX analysis of titanium-rich phase (C) in red mud



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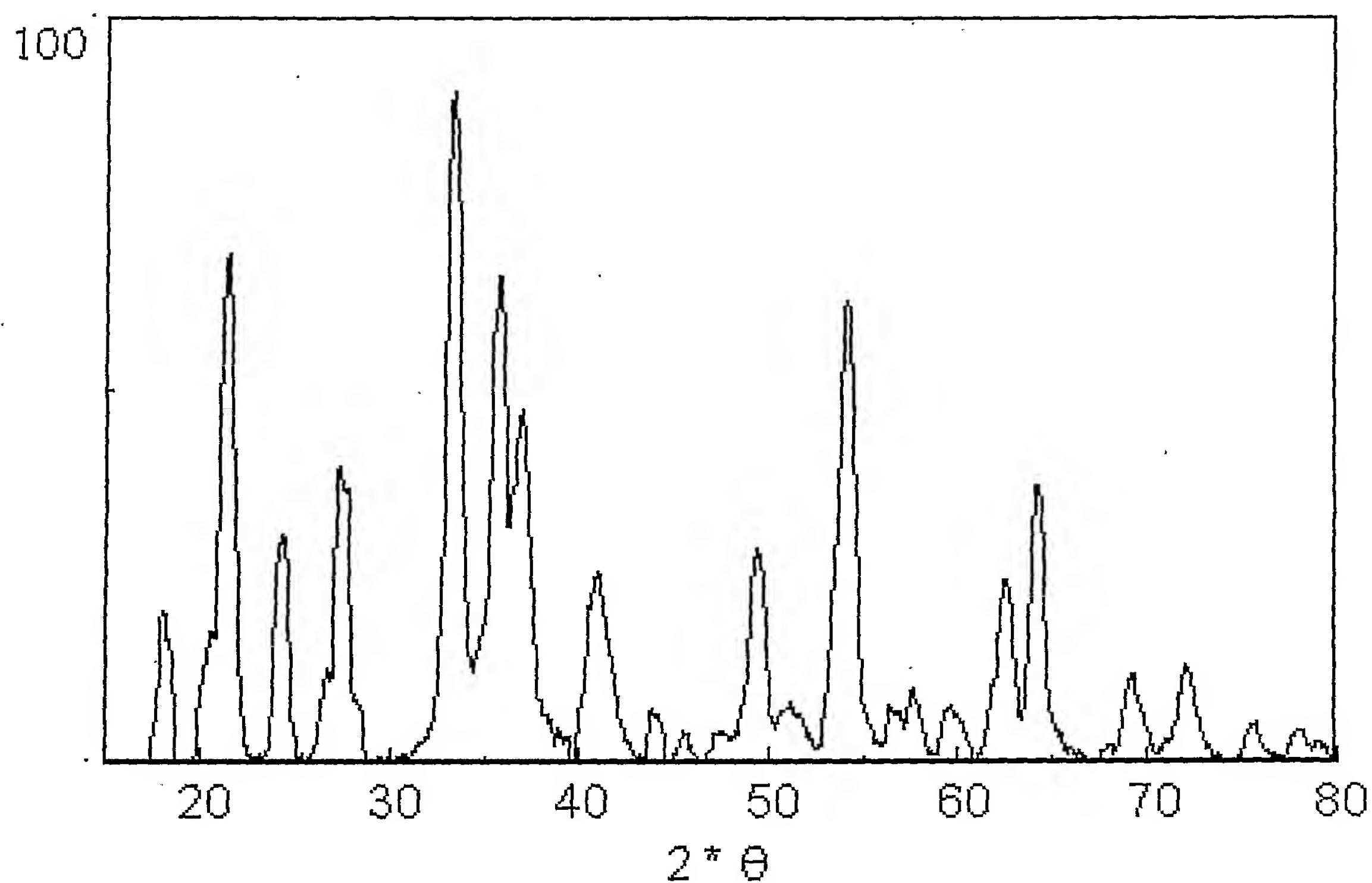


Fig. 5

X-Ray diffraction pattern of red mud

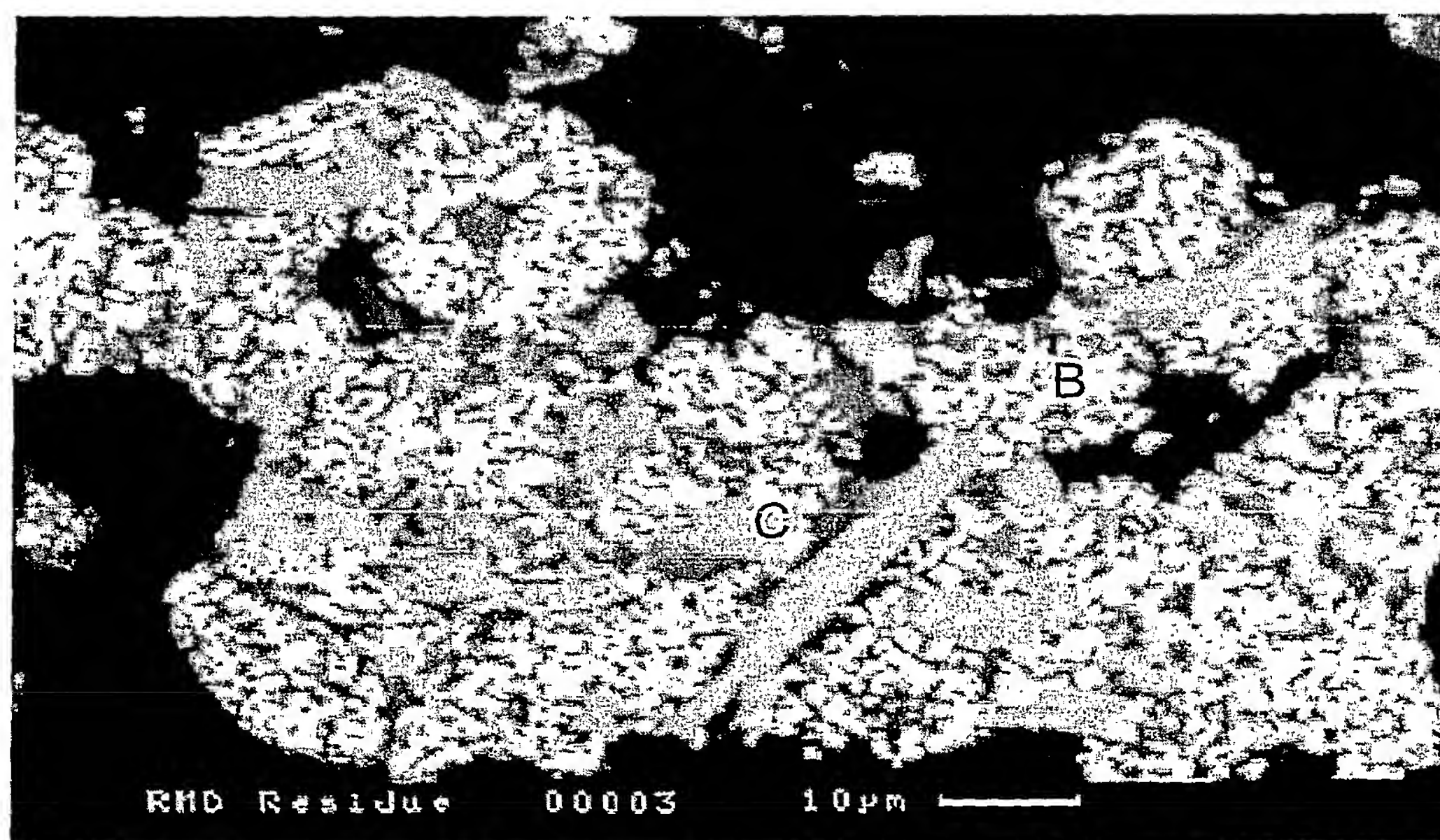


Fig. 6

Micrograph of residue after extraction of alumina from red mud.  
B : Iron rich phase C : Titanium rich phase

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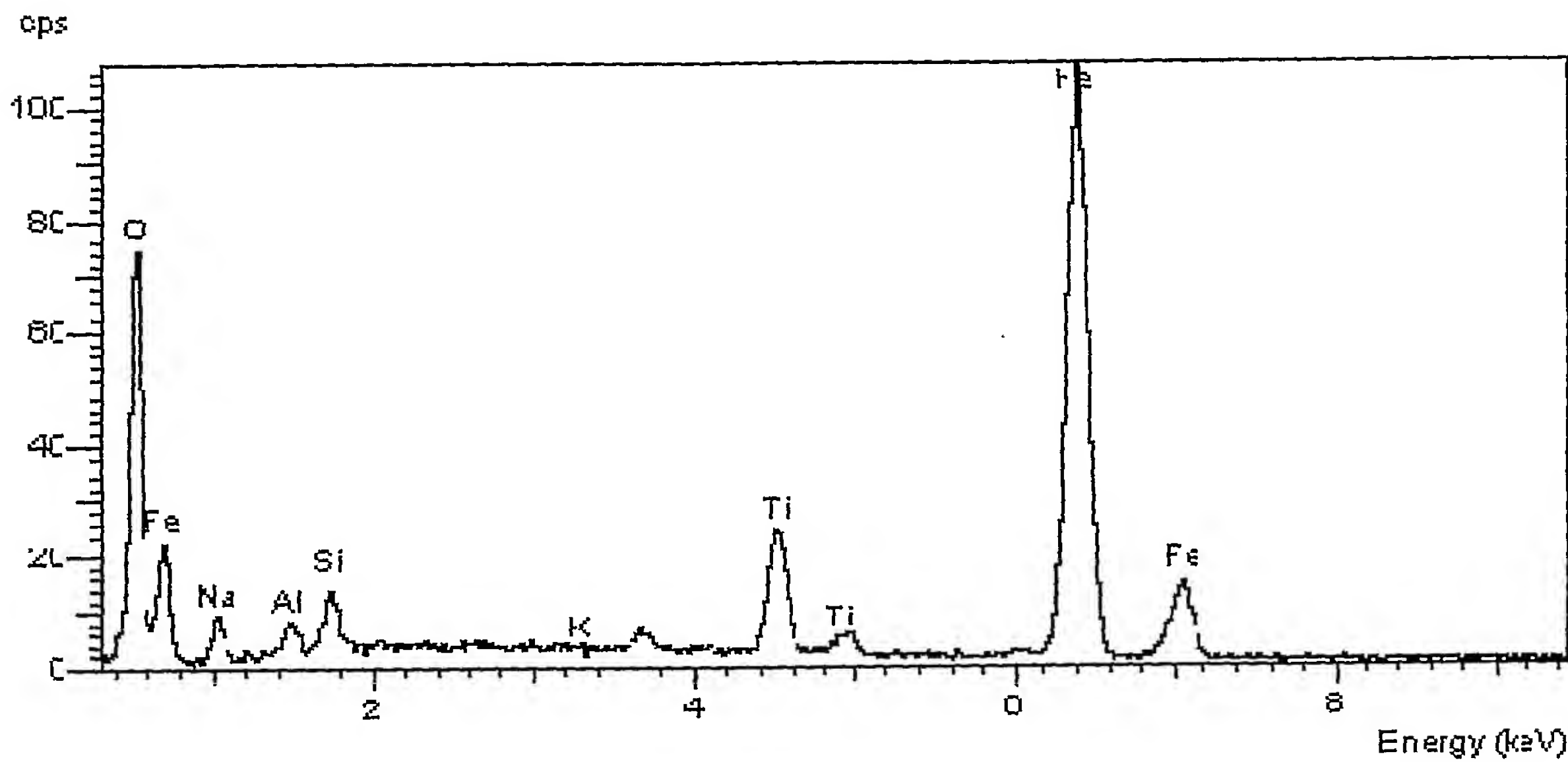


Fig. 7

EDX analysis of iron-rich phase (B) in the residue after extracting alumina from red mud

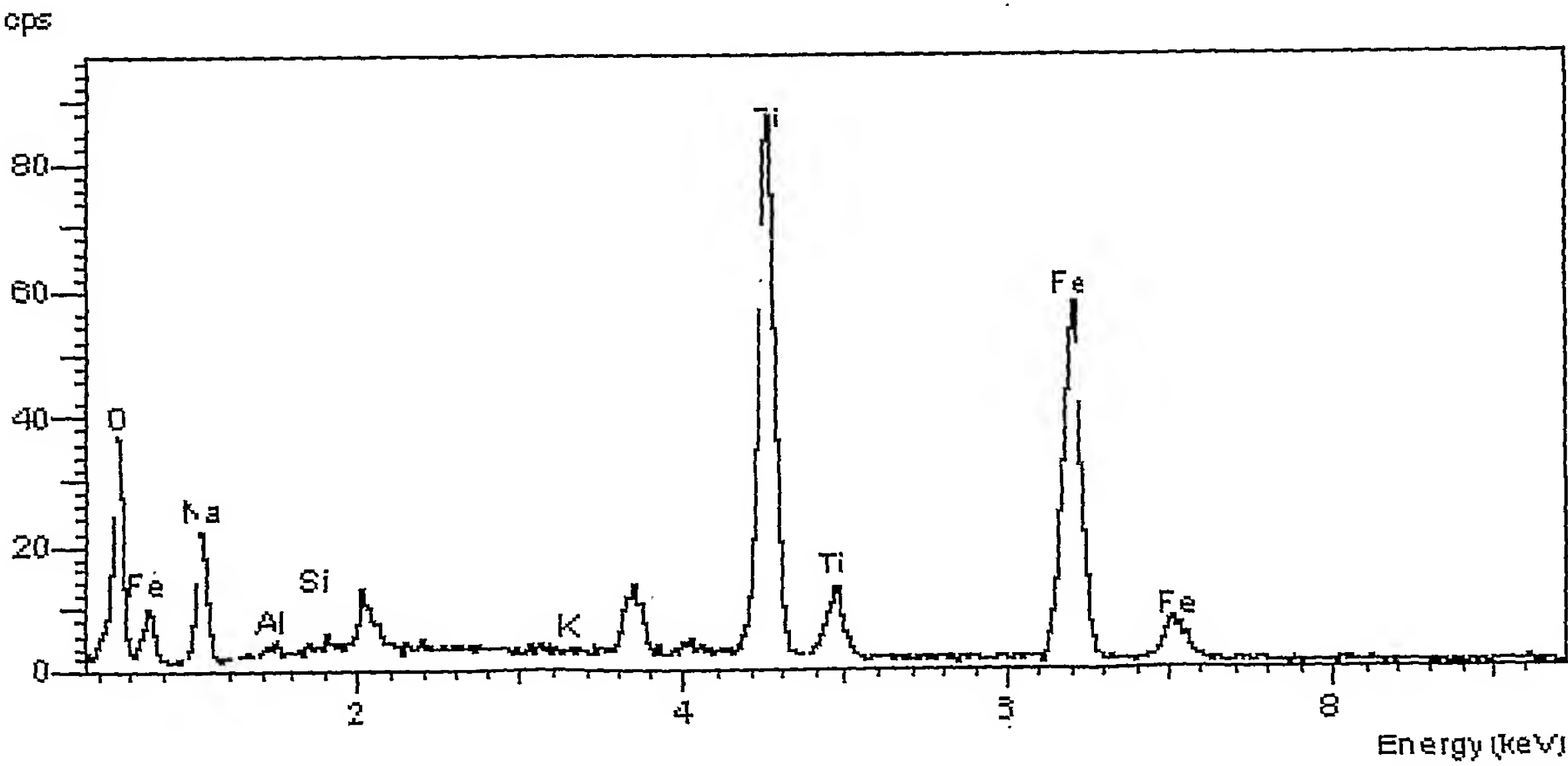


Fig. 8

EDX analysis of titanium-rich phase (C) in the residue after extracting alumina from red mud

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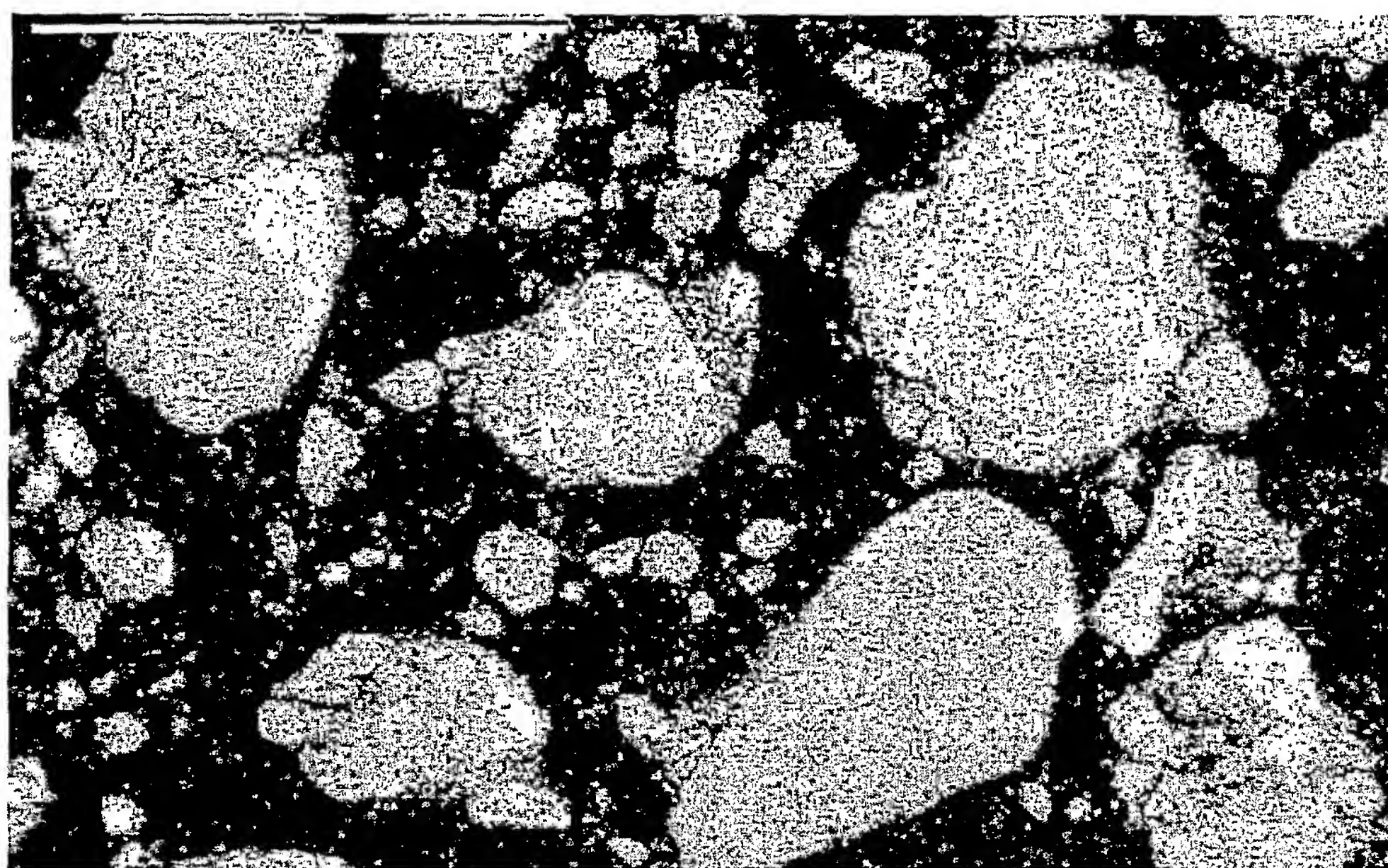


Fig. 9(a)

Micrograph of Alumina extracted from red mud

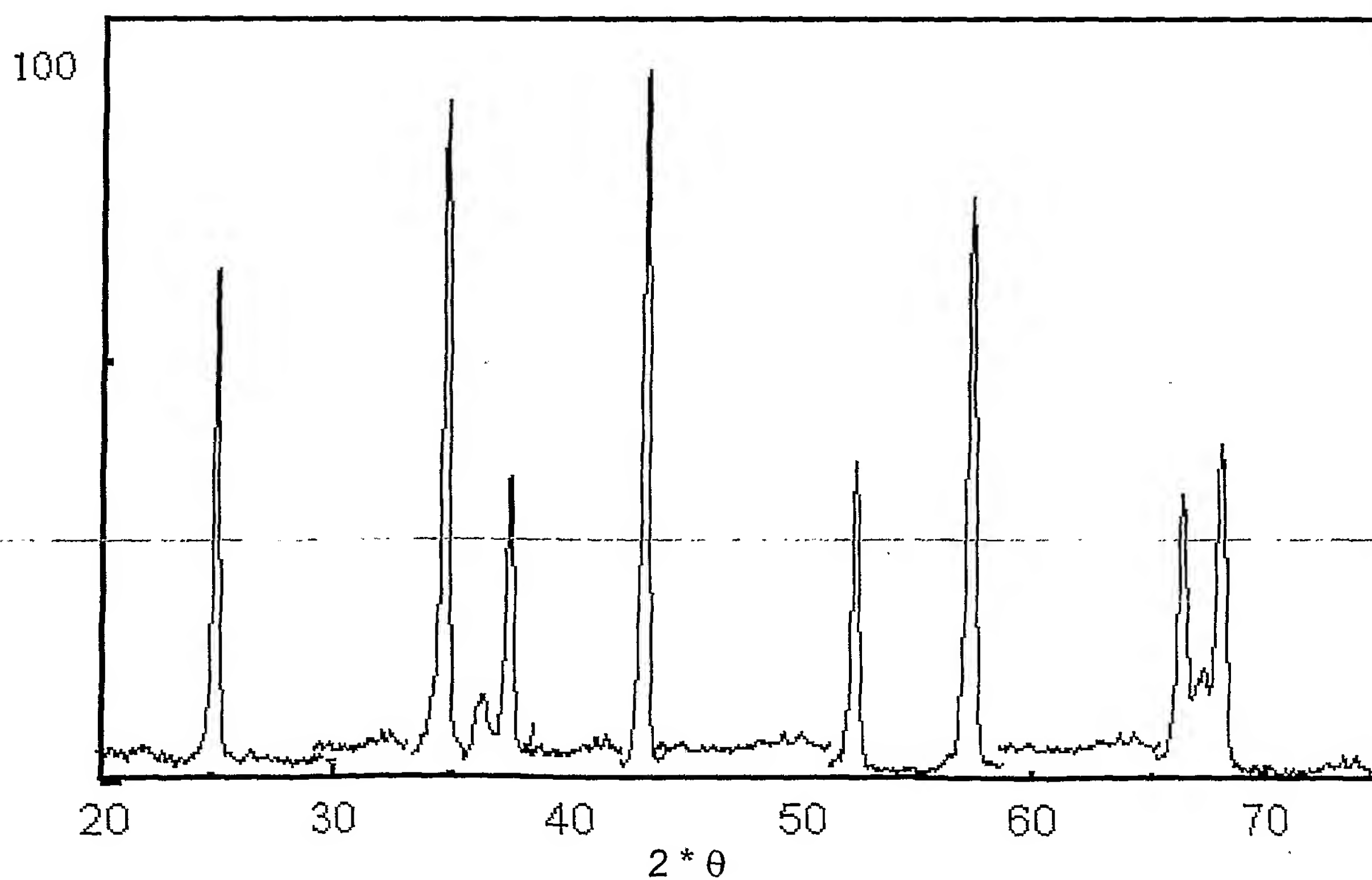
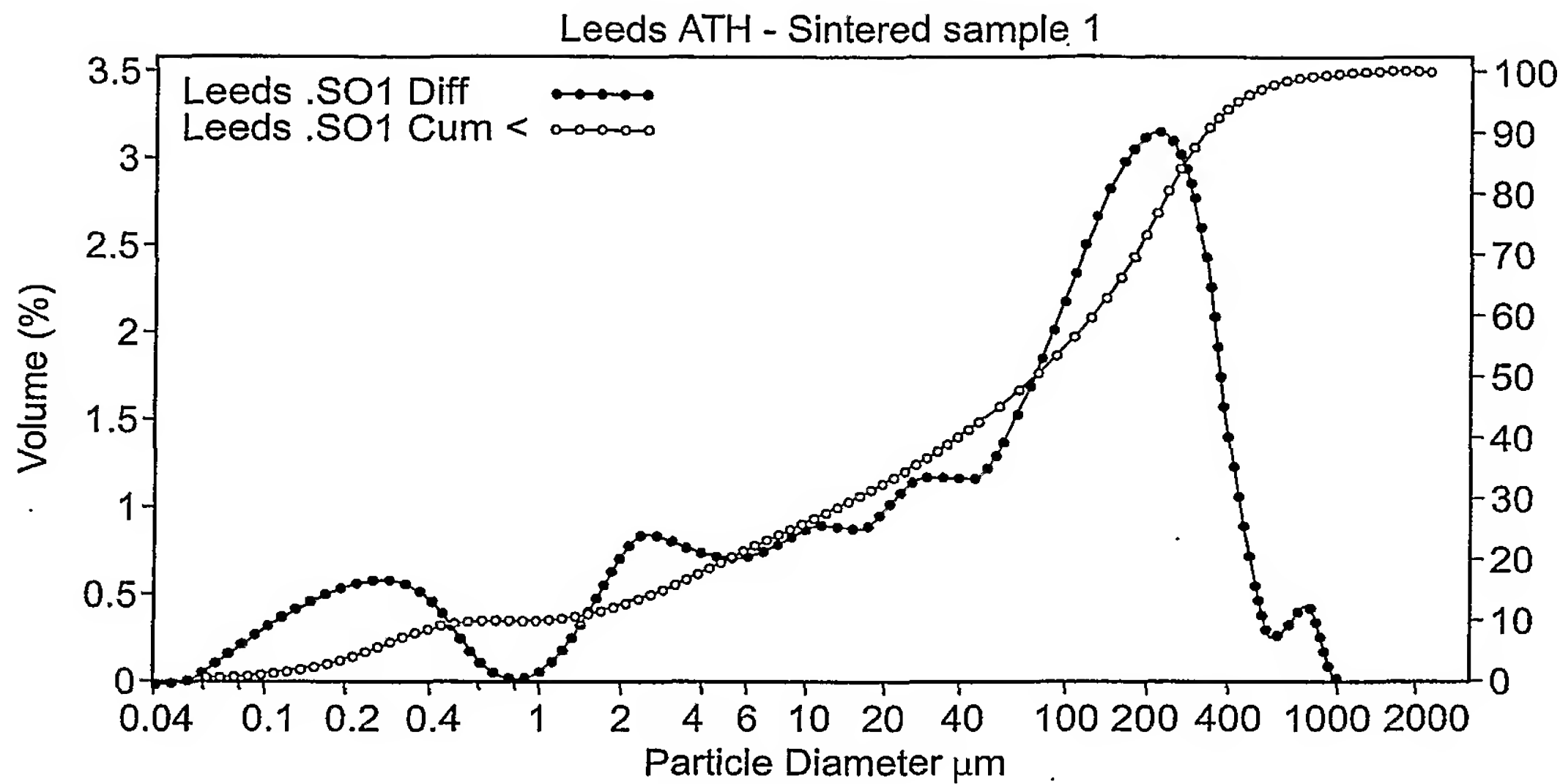


Fig. 9(b)

X-Ray Diffraction pattern of Alumina extracted from red mud

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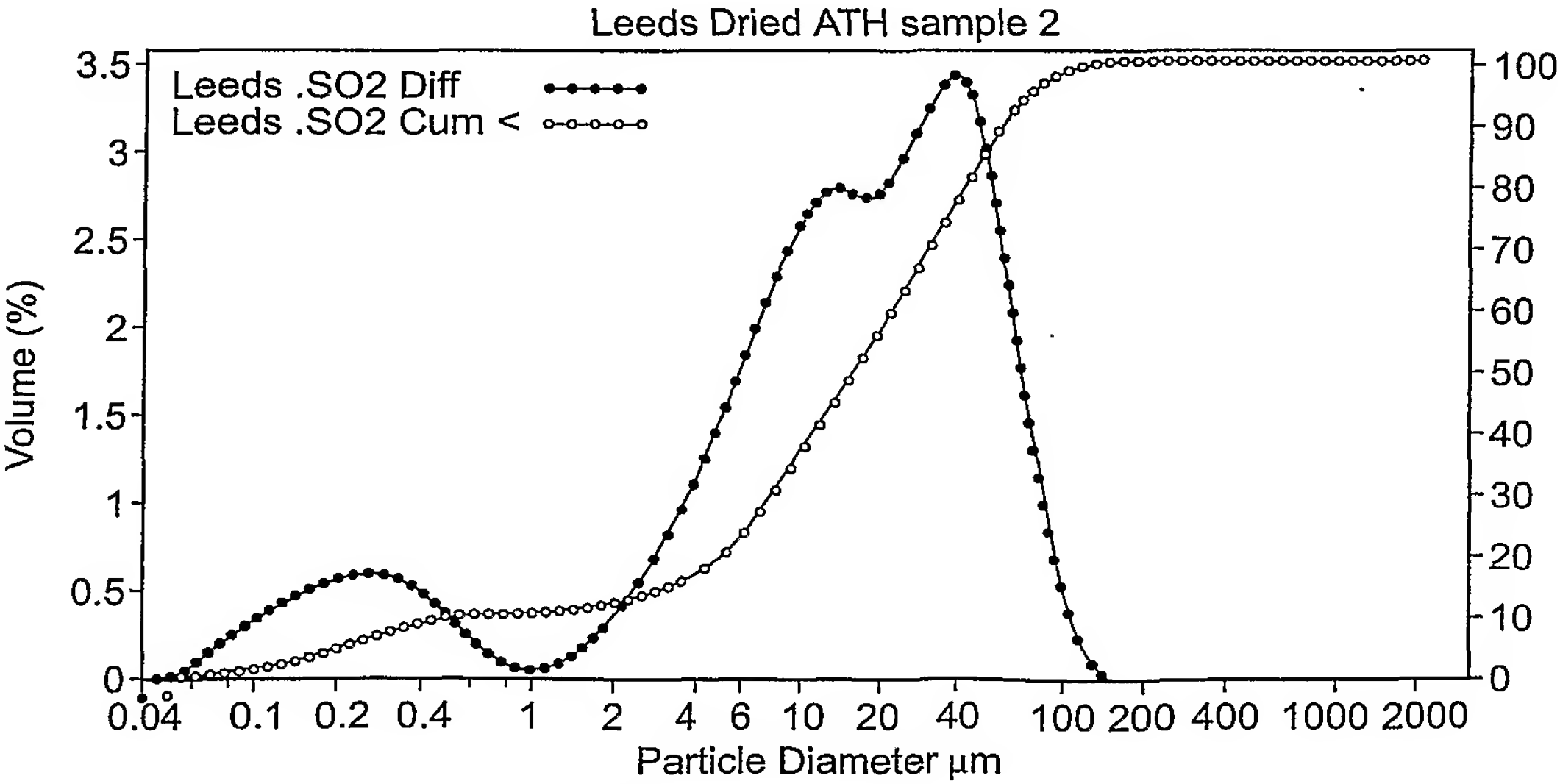


Volume Statistics (Arithmetic) Leeds SO1

Calculations from 0.400  $\mu\text{m}$  to 2000  $\mu\text{m}$

Volume:	100%				
Mean:	124.1 μm		S.D.:	145.7 μm	
Median:	78.98 μm		C.V:	117%	
D(3,2):	1.707 μm				
Mode:	203.5 μm				
%<	10	25	50	75	90
μm	1.297	9.773	78.98	191.6	305.5

Fig. 10(a)  
Particulate size distribution  
of  $\text{Al}(\text{OH})_3$  after drying



Volume Statistics (Arithmetic) Leeds SO2

Calculations from 0.0400  $\mu\text{m}$  to 2000  $\mu\text{m}$

Volume:	100%				
Mean:	22.79μm		S.D.:	21.94 μm	
Median:	15.84μm		C.V:	94.9%	
D(3,2):	1.550μm				
Mode:	34.58 μm				
%<	10	25	50	75	90
μm	0.573	0.323	15.84	34.26	53.38

Fig. 10(b)  
Particle size distribution  
of alumina after calcining  
at 1400K



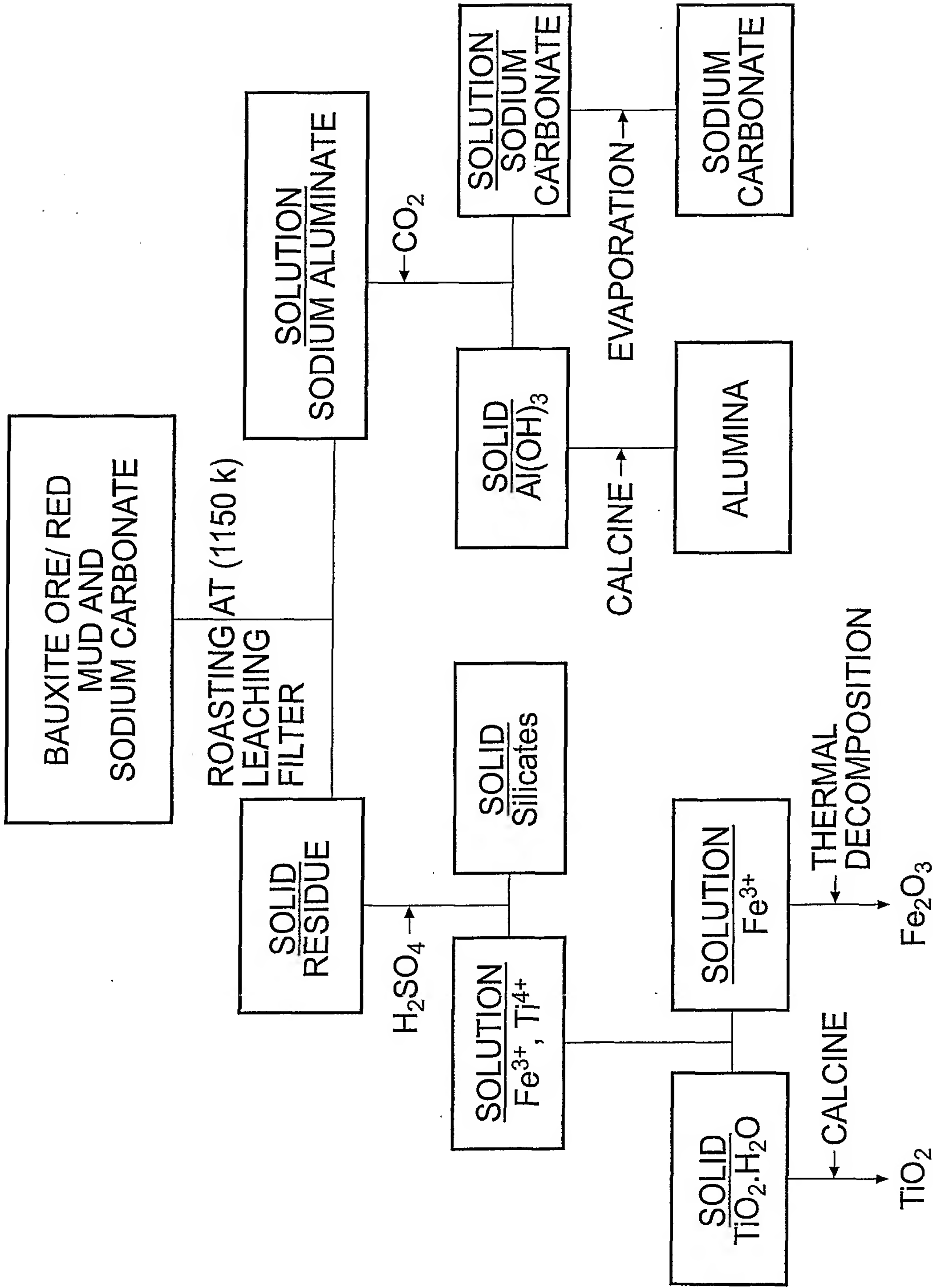


Fig. 11



## INTERNATIONAL SEARCH REPORT

International Application No

PCT, GB 01/03370

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C01F7/08 C01G23/047 C01G49/06

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C01F C01G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4 254 088 A (MCDOWELL WILLIAM J ET AL) 3 March 1981 (1981-03-03) the whole document ---	1-29
X	GB 848 230 A (BLANCS DE ZINC DE LA MEDITERRA) 14 September 1960 (1960-09-14)  examples 1,3 ---	1-5, 7-13, 15-20, 23-26, 29
X	GB 601 968 A (MONOLITH PORTLAND MIDWEST COMP) 18 May 1948 (1948-05-18)  the whole document --- -/--	1-3, 10-13, 15-18, 20, 23-26, 29



Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

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- \*P\* document published prior to the international filing date but later than the priority date claimed

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- \*X\* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
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- \*&\* document member of the same patent family

Date of the actual completion of the international search

6 November 2001

Date of mailing of the international search report

14/11/2001

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# INTERNATIONAL SEARCH REPORT

In            onal Application No  
F 01, GB 01/03370

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4 265 864 A (SUGAHARA YUJIRO ET AL) 5 May 1981 (1981-05-05) examples 1,3 ---	1-7, 10-13,29
A	US 4 119 698 A (ZIMMER ERICH ET AL) 10 October 1978 (1978-10-10) the whole document -----	1-29

INTERNATIONAL SEARCH REPORT

Information on patent family members

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PCT/GB 01/03370

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